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SURFACE RESPONSE METHODOLOGY FOR THE COAGULATION OF SOLID PARTICLES IN COAL EFFLUENT USING CHITIN-DERIVED COAGULANT

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ABSTRACT
The use of Chitin-derived coagulant (*Chitosan*) in the removal of solid particles from coal effluent was examined in this study. Coagulation-flocculation was conducted at the pH levels of 2 (acidic), 6 (less acidic) and 10 (basic), and also at various dosages of 100, 200 300, 400 and 500mg/l of the coagulant. The Jar Bench – scale tests showed that particle removal increases with time. Very high coagulation rate was observed at pH of 6 (99.95% efficiency), followed by that at pH of 10 (99.5% efficiency), and least at pH of 2 (90.5% efficiency). Coagulation capacity was, also, found to increase as the coagulant dosage increases. 2^3-full factorial design was employed to study the surface response/ behaviour of the experimental samples, in which three factors (*Dosage, pH and Contact duration*) were considered. Design Expert software (7.0 version) was used in plotting the factor effects (singlet and interactions), which were subsequently utilized in studying the design characteristics for a possible optimal approximation. The plots of single factor effect showed that particle concentration reduces as the stirring time and pH increase, but increases at higher dosages. The interactions between Dosage and pH, as well as that between Dosage and Stirring time showed significant difference (statistically), unlike in the case of pH and Stirring time interaction. However, a very high Particle removal from the effluent sample was achieved by going to high pH of 10 and high stirring time of 30minutes, and this was found to be the optimal point.

KEYWORDS: Surface Response, Coagulation, Solid Particles, Coal Effluent, Chitosan

INTRODUCTION

Surface Response Methodology (SRM) explores the relationships between several explanatory variables and one or more response variables. The method was introduced by G.E.P. Box and K.B. Wilson in 1951 (Wikipedia, 1990). According to the review, the main idea of *SRM* is to use a set designed experiments to obtain an optimal response. It was acknowledged that this model is just an approximation, but was used because such a model is to estimate and apply, even when little is known about the process. An easy way to estimate a first-degree polynomial model is to use a *factorial experiment* (*which can be either a full factorial design or a fractional factorial design*). This is sufficient to determine which explanatory variables have an impact on the response variable(s) of interest. Once it is suspected that only significant explanatory variables are left, a more complicated design, such as a Central Composite Design, can be employed to estimate a second-degree polynomial model, which is still only an approximation at best. However, the second-degree can be used to optimize (minimize, maximize, or attain a specific target for) a response.

Some extensions of surface response methodology deal with the multiple response problem. Multiple response variables create difficulty because what is optimal for one response may not be very optimal for other responses. Other extensions are used to reduce variability in a single response while targeting a specific value, or attaining a near maximum or minimum outcome while preventing variability in that response from getting too large. Significant criticisms of *SRM* include the fact that the optimization is almost always done with a model for which the coefficients are estimated, not known. That is, an optimum value may only look optimal, but be far from the truth because of variability in the coefficients. Brathy (1998) reported that the tests used for optimization studies are usually from design of experiments (*DOE*) fields. According to the report, the *DOE* involves the design of all information-gathering exercises where variation is present, whether under full control of experiment or not. A contour plot is always used to find the responses of two variables by including a large number of trials in each and every combination of them, and using some sort of interpolation to find potentially better intermediate values between them. But since experimental runs often cost a lot time and money, it may be difficult to pinpoint the ideal coefficients. In other words, when one needs to screen a larger number of factors in

order to identify those that may be important (that is those that are related to the dependent variable of interest), it is important to employ a design that allows for testing of largest number of factor main effects with least number of observations. However, there are standing strategies used to find those values with minimal runs; the present study explores experimental design in *SRM* to make necessary tradeoffs between reducing variability and reducing any intending negative impact in the coagulation of solid particles from coal effluent using Chitin-derived coagulant.

MATERIALS AND METHODS

Sample Collection: The wastewater sample was collected from Akwukwe Coal Mining industry, Enugu-Nigeria. The *TSP* removal from coal washery effluent was investigated in the laboratory using coagulation-flocculation techniques. The coagulant used-*(Chitosan)* was bought from *'Eke-ukwu Owerri'* local market in Imo State, Nigeria.

pH Measurement: Cyberscan-510 pH meter was used in determination of the pH. The instrument was first calibrated with known pH solution. Two-point calibration method was adopted in which two buffers of pH – 4.0 and 7.0 were selected for calibration. The first buffer selected was near the isopotential point and the second near the expected pH of the sample. For cross-checking of the instrument, a buffer solution of known pH was measured by dipping the probe in the sample. The probe was constantly stirred until a stable pH reading was obtained.

Total Solid Particles (TSP): Total solid was measured by oven-drying method. The sample was taken in a sizeable evaporating dish and dried at controlled temperature of 103-105^{o}C for 1 hour, and is cooled in a desicator to a favourable temperature. The 250-300ml of unfiltered well-mixed sample was taken in a beaker and put in the air-oven at 103-105^{o}C for 2 hours and weighed after cooling. The process was repeated until a constant weight was obtained.

$$TSP\ (mg/l) = (W_f - W_i) * 1000/Volume\ of\ Sample\ (mg/l)$$

Where, W_f = final weight of beaker (mg)
W_i = initial weight of beaker (mg)

Jar Tests: The coagulation experiments were conducted with the Chitin-derived coagulant (*Chitosan*), using six jars and multiple stirrers, with 10 minutes rapid mixing and 20 minutes slow mixing. This was followed by 30 minutes of settling. Also, settling tests were conducted for determination of settling behaviour of the effluent after the addition of each *CTS*-coagulant dose. Effluent sample was taken in 1 litre measuring cylinder and the required coagulant dosages were added and stirred properly. Settling rate of floc with respect to time was measured; after 30 minutes settling, sludge volume was, also, measured in terms of height.

The Factorial Experiment

Coagulation-flocculation experiments were conducted randomly at two different levels – *the High level* (+1) and *Low level* (-1) as contained in table 1, with reference to three experimental factors – *Coagulant dosage* (A or x_1), pH (B or x_2) and *Stirring time* (C or x_3). Initial effluent concentration was checked by means of Ultraviolet Spectrophotometer (UVS) before the process. The finally concentration was, also, examined after every random treatment, and this served as response for the design process. Eight different experiments were conducted using the design array developed by Frank Yates (Wikipedia, 1990), with replications to ascertain homogeneity of the experimental method.

Table 1: BASIS FOR THE DESIGN EXPERIMENTS

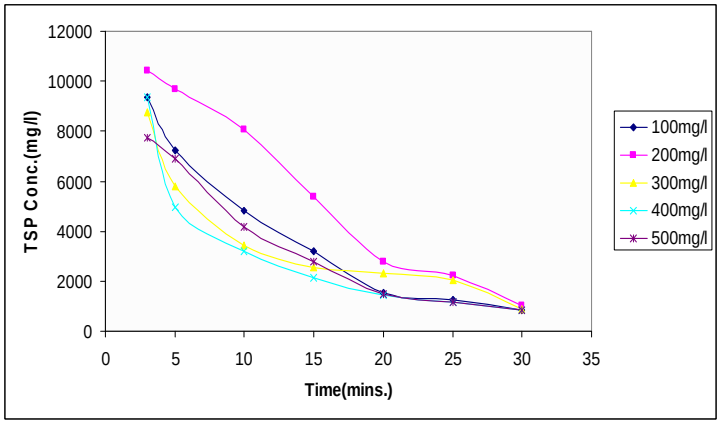
Factors	High Level (+)	Low Level (-1)
CTS Dosage, x_1(mg/l)	500	100
pH of Solution, X_2	10.0	2.0
Contact Time, X_3(mins.)	30.0	5.0

RESULTS AND DISCUSION

The surface responses for the factorial experiments (y_1) and their corresponding replications (y_2) are presented in table 2. The jar bench-scale results (Fig. 1-3) showed that the amount of solid particles (which is measured as

the Total Solid Particles – *TSP*) removed increases with time. In other words, the highest amount of particles was removed at 30 minutes (within the time frame studied). This explains that as time proceeds, more destabilization of particles occurs, which leads to greater particle growth through settling. The efficiency plots (Fig. 4-6), also, show that there was a substantive solid particle removal within the initial stages of the process. This may be traced to the sharp rise in the Efficiency-Time profile within the first 10 minutes. It may be observed that the coagulation capacity increases as the coagulant dosage is increased. This suggests that increase in coagulant dosage favours the rate of coagulation process.

The plots of *'one factor effect'* of the design factors (Fig 7-9) showed that particle concentration reduces as the stirring time and pH increase, but increases at higher dosages. It may, also, be observed that there is significant difference (statistically) between factor-A (dosage) and factor-B (pH), as well as between factor-A (dosage) and factor-C (stirring time). This is demonstrated by the overlapping intervals in the interaction plots (Fig 10 and 11), unlike in the case of factors-B and C that did not overlap (Fig 12). From the points of overlap in figures 10 and 11, it could be observed that the spread of data points is less at the right-hand side (where A is high) than the left-hand side (where A is low). This implies that the effect of pH and stirring time is less significant at high dosages, even though the particle concentration is reduced. Also, the appearance of the number-*2* beside some data points in figures 11 and 12 indicates the presence of multiple points at the same location; clicking at the points in the Design Expert windows displays the number of data points contained in the location. The 3-D surface plots (Fig 13-15) showed similar trend as the other *FD*-plots; the concentration was least at pH of 10 and dosage of 300mg/l for Fig 13, and TSP removal was insignificant at 500mg/l dosage (*as indicated by the disappearance of the LSD-bar at this dosage*). Similarly, concentration was least at the stirring time of 30 and dosage of 100mg/l for Fig 14, while it was least at stirring time of 30 and pH of 6 for Fig 15. It, now, becomes clear that a very high TSP removal from the effluent sample could be achieved by going to high pH (unlike in the normal coagulation process, where coagulation efficiency was highest at pH of 6 as indicated in Fig 5) and high stirring time at *q*mg/l dosage, such that: $100 \leq q \leq 300$; this is the optimum outcome, and for this study, it is located at *pH of 10, stirring time of 30mins* and *dosage of 300mg/l*.



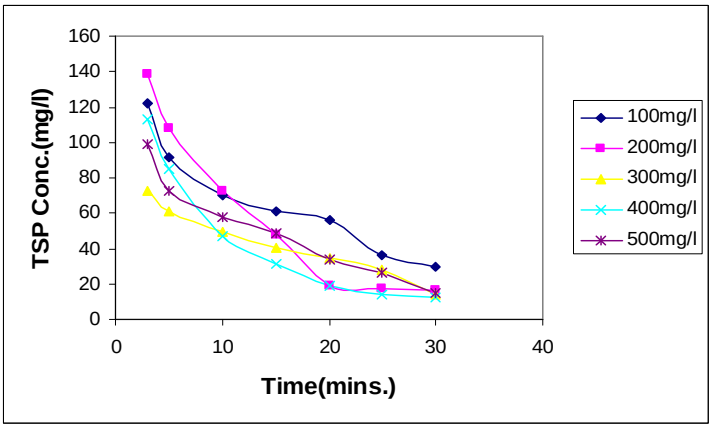
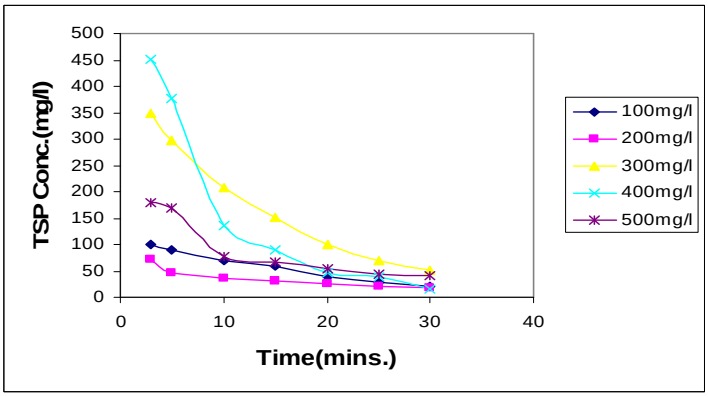


Fig 1:Conc.-Time Plot at pH of 2 (C_o=16074.00mg/l); Fig 2:Conc.-Time Plot at pH of 6 (C_o=23171.00mg/l)



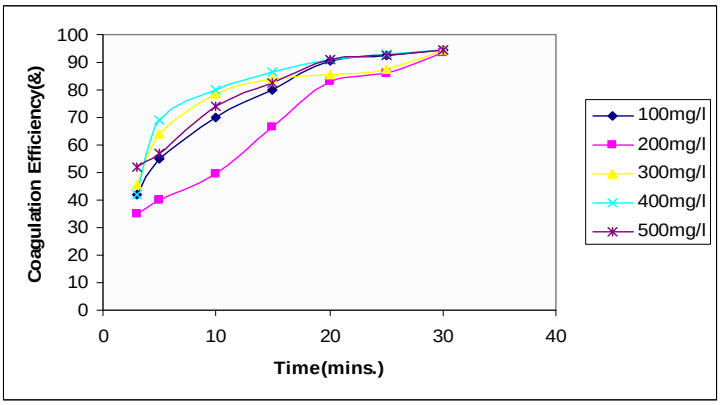
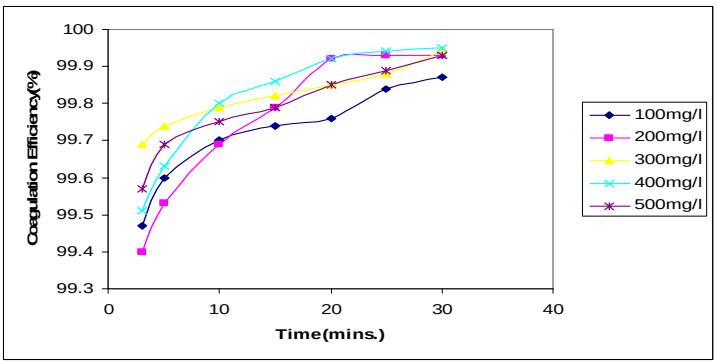


Fig 3:Conc.-Time Plot at pH of 10 (C_o=16074.00mg/l); Fig 4: Coagulation Efficiency for CTS Doses at pH of 2



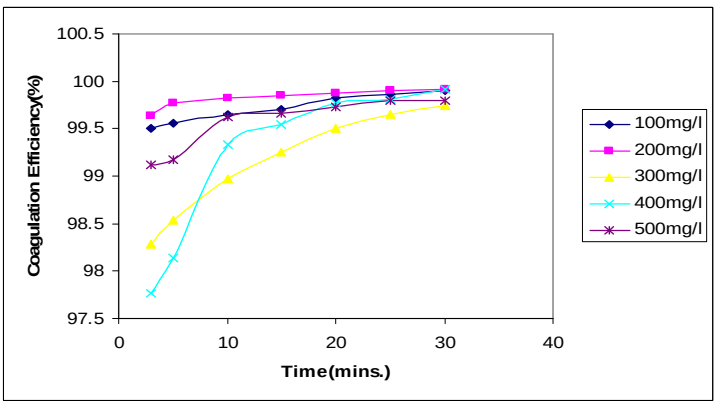


Fig 5: Coagulation Efficiency for CTS Doses at pH of 6; Fig 6: Coagulation Efficiency for CTS Doses at pH of 10

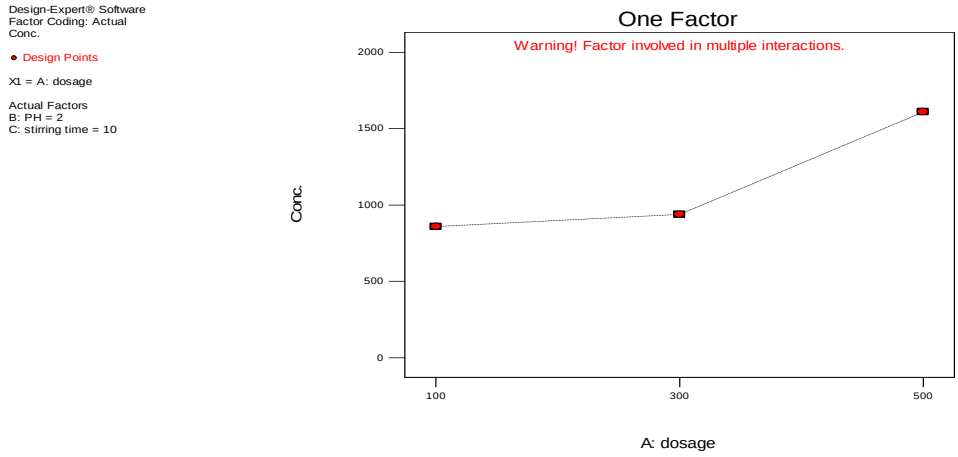


Fig 7: Effect of Factor-A (Dosage) on Particle Concentration

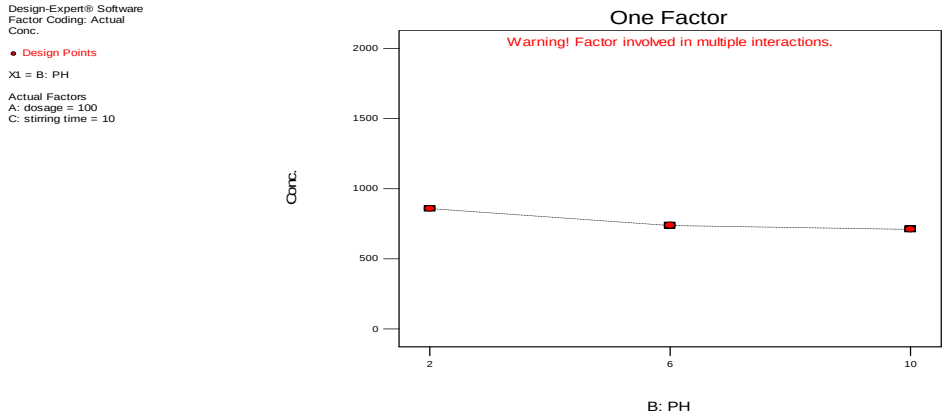


Fig 8: Effect of Factor-B (pH) on Particle Concentration

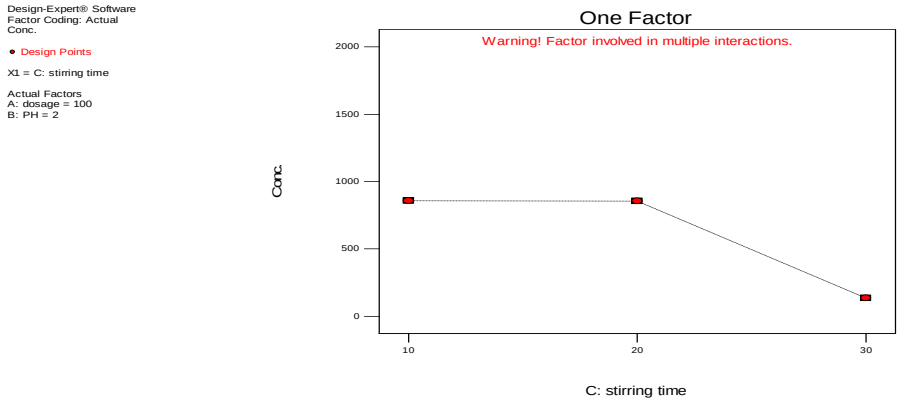


Fig 9: Effect of Factor-C (Stirring Time) on Particle Concentration

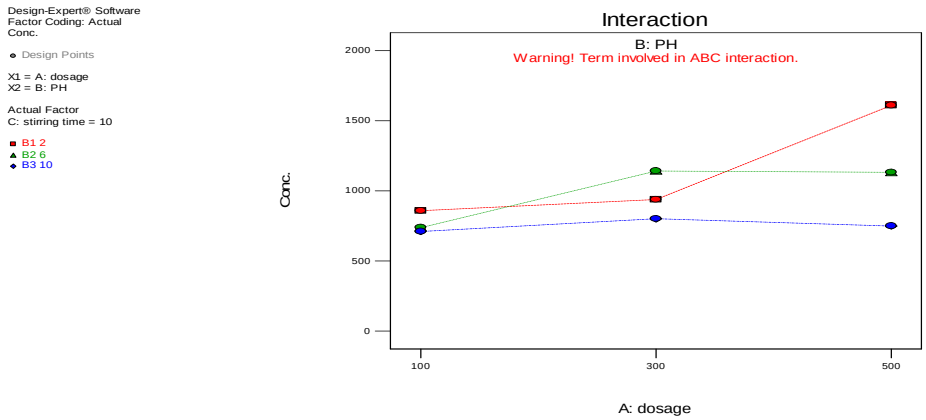


Fig 10: Effect of Interaction between Factors A and B on Conc.

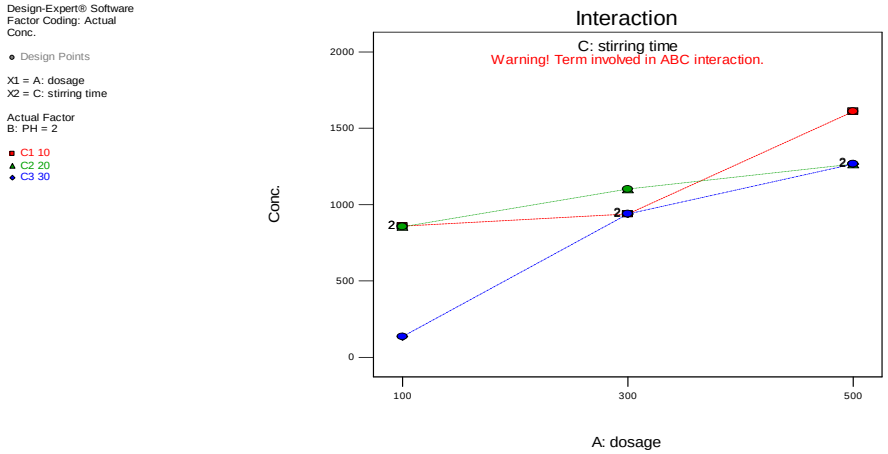


Fig 11: Effect of Interaction between Factors A and C on Conc.

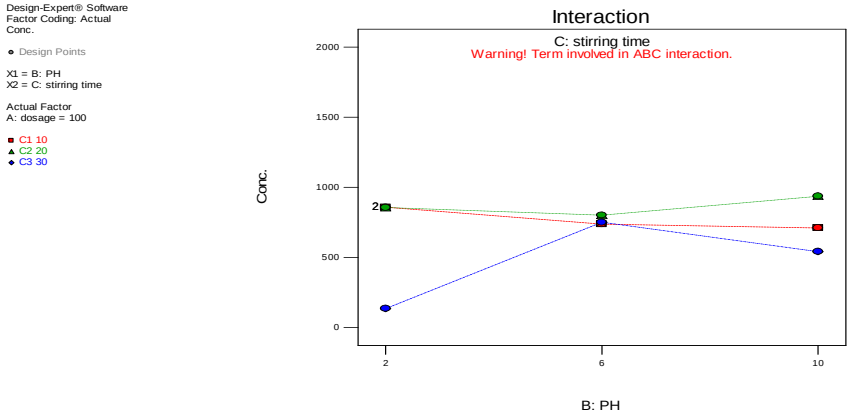


Fig 12: Effect of Interaction between Factors B and C on Conc.

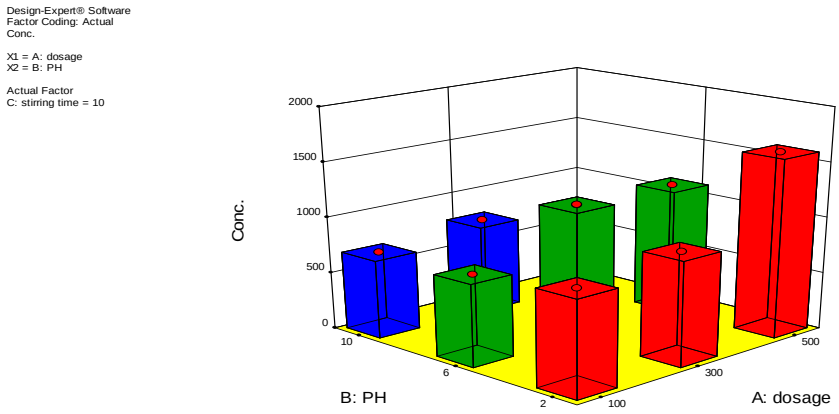


Fig 13: 3-D Surface Plot for Dosage (A) Versus pH (B)

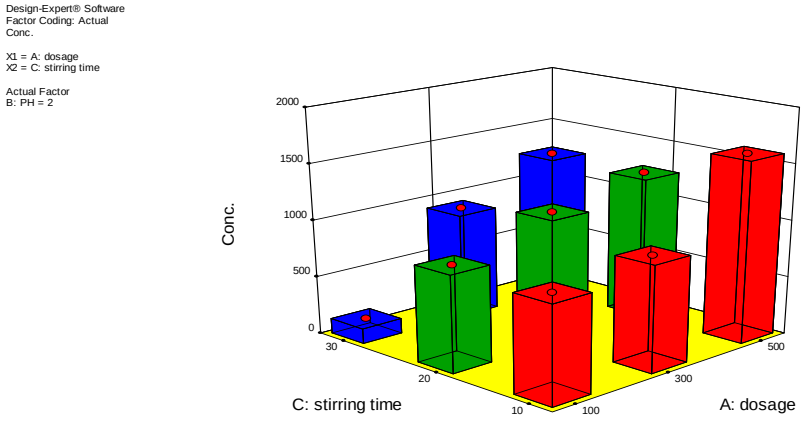


Fig 14: 3-D Surface Plot for Dosage (A) Versus Stirring Time (C)

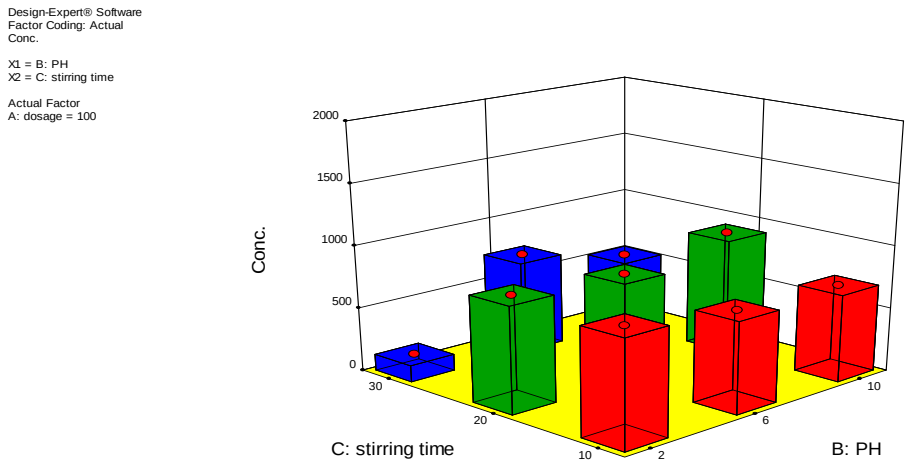


Fig 15: 3-D Surface Plot for pH (B) Versus Stirring Time (C)

Table 2: SURFACE RESPONSES FOR THE DESIGN FACTORS

No of Runs	X_1(mg/l)	X_2	X_3(mins.)	Y_1(mg/l)	Y_2(mg/l)
1	100, -1	2.0, -1	5.0, -1	7726.8	7341.5
2	500, +1	2.0, -1	5.0, -1	6817.25	6034
3	100, -1	10.0, +1	5.0, -1	4159.5	4326.5
4	500, +1	10.0, +1	5.0, -1	2773.1	2580.55
5	100, -1	2.0, -1	30, +1	1461.7	967.40
6	500, +1	2.0, -1	30, +1	1169.36	1082.71
7	100, -1	10.0, +1	30, +1	853.05	851.94
8	500, +1	10.0, +1	30, +1	801.35	794.38

CONCLUSION

Each of the random experiments has a definite amount of measurable conversions, which are manifested in the respective responses. This ensured that there was enough data for a thorough statistical evaluation. Since full factorial testing requires that every possible experimental combinations (interaction of factors) is shown, *'our 3-factor'* test critically displayed the whole eight(8) experiments (2^3 – experiments), together with their replications. Randomized factor interactions at varying levels of relevance employed showed that the optimal rate of particle removal from the effluent sample falls at the pH of 10, *CTS* dosage of 300mg/l and Contact time of 30minutes under the boundaries of the factors studied.

NOMENCLATURE

CTS – Chitosan
SRM – Surface Response Methodology
DOE – Design of Experiment
TSP – Total Solid Particles
FD – Factorial Design
LSD – Least Significant Difference

REFERENCES

Box, B.E.P. and Wilson, K.B. (1951) Experimental Attainment of Optimum Conditions (with discussion) . *Journal of the Royal Statistical Society* . Series B 13(1):1-45

Brathy, J. (1998) Design of Flocculation Systems from Batch Test. *Journal water Sciences*, 3(4), pp 173-182.

Dick, R.I. and B.B. Ewing (1997) Evaluation of Activated Sludge Thickening Theories. *Journal of American Society of Civil Engineers*. 93(4), pp 41-53.

John L. R. (2001) Chemical Process Design and Analysis. Burns and Roe Inc., New Jersey. pp 43-50.

Maiti, S.K. (2006) Study on settling Behaviour of Coal Washery Effluent-A Case Study. Proceedings of the National Seminar on Environmental Engineering. Centre for Mining Environment, Dhanbad-India.

Maiti, S.K. (2001) Handbook of Methods in Environmental Studies, Vol. 1-ABD Publishers, Jaipur.

Menkiti, M.C., P.K. Igbokwe, F.X.O. Ugodulunwa and O.D. Onukwuli (2008) Rapid Coagulation/Flocculation Kinetics of Coal Effluent with High Organic Content Using Blended and unblended Chitin Derived Coagulant (CDC). *Research Journal of Applied Sciences*, 3(4), pp 317-323.

Perry, R.H., D.W. Green and J.O. Maloney (1984) Perry's Chemical Engineer' Handbook, 6th ed. McGraw-Hill Company Ltd, New York.

Rose, J.B. and C.P. Garba (1991) Assessing Potential Health Risks from Viruses and Parasites in Reclaimed Water, In: *Water Science Technology*. *Journal of U.S. EIA*, 23(4), pp 470-493.

Shilpi Kushwaha (2003) Equilibrium, kinetics and Thermodynamic Studies for Adsorption of Hg (II) on Palm Shell Powder. *Journal of World Academy of Science, Engineering and Technology*, 43(4), pp 600-605.

Sinnott, R.K. (1999) Coulson and Richardson's Chemical Engineering . Vol. 6 . Butterworth Heinmann, Oxford.
Treybal, R.E. (1981) Mass-Transfer Operations . McGraw-Hill Inc, New York

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